LALP-07-054 Summer 20

# Materials science and engineering from start to finish

# **Sigma Complex**



Unique composition niobium-silicide alloy, plasma melted for the Air Force as part of advanced engine development.



Hot and cold rolling of metals is used to meet customer dimensional and crystallographic requirements.

he original and enduring mission of the Sigma Complex focuses on prototype fabrication and materials research for the nuclear weapons program. Over the years, however, this mission has expanded to include threat reduction and homeland security activities.

Today, the Sigma Complex supports a large, multidisciplinary materials technology base. Research and development activities include a synergistic blend of metallurgy and ceramics focused on the integration of synthesis and processing, characterization, and fabrication combined with fundamental computer modeling and process simulation.

These extensive capabilities reflect significant investments in metal casting; thermomechanical processing; electrochemistry; powder processing; sol gel synthesis and colloidal processing; laser and microwave processing; process modeling; and microstructural, mechanical, and thermophysical properties characterization.

The Sigma Complex is unique at the Laboratory—and perhaps within the United States—because this capability investment provides a highly integrated approach where materials can be

processed on a large scale from the raw state to a final product. In this facility ceramists and metallurgists work with engineers and physicists to solve mutual problems and to extend understanding of materials processes and applications.

Highly qualified personnel and capability investments have maintained state-of-the art technology to create a responsive infrastructure that can quickly respond to customers' needs. The facility is ideally suited for rapid turnaround of small lot production and prototype development. Several collaborations between Los Alamos and other Department of Energy (DOE) complex sites utilize Sigma's unique capabilities to solve design and production issues across the nuclear weapons programs. For example, Los Alamos is assisting Y-12 with welding and casting development to improve production quality and efficiencies.

Our unique machining capabilities are routinely used by customers throughout the Laboratory and the DOE complex. The expertise within Sigma is regularly used to evaluate materials issues for providing input to re-use/remanufacture decisions and to develop alternative materials and processing methods for the nuclear weapons programs.





An aluminum instrument component for the International Border Explorer (IBEX) satellite is given three different coatings to yield a radiation absorbent black copper oxide surface. This unique coating process was developed at Los Alamos National Laboratory.



#### **Materials Science and Technology Division Facility Focus**

### **Prototype Fabrication** and Small Lot Manufacturing

The Sigma Complex represents an advanced manufacturing research facility with unique capabilities to improve manufacturing and fabrication processes and to produce prototype components in support of the Laboratory's stockpile stewardship programs. Most teams within MST-6 contribute to this effort including the Beryllium Team, the Foundry-Machining Team, the Welding and Joining Team, and the Corrosion and Electrochemistry Team.

Some of the fabrication methods available at the Sigma Complex include: multi-axis computer numerically controlled/ electric discharge machining (CNC/EDM); casting, rolling and forging; hydroforming, electroforming, swaging and drawing; heat-treating, high-energy density welding, such as electron beam welding and laser beam welding (both continuous wave and pulsed), precision arc welding, brazing, plasma spraying, and near net shape (NNS) hot isostatic pressing.

Projects include more than original fabrication. Objects are re-created for customers using a coordinate measuring machine (CMM) and CNC. Declassification of classified metal parts by melting and machining is an important contribution to security.

#### **Recent projects**

- **Pit Manufacturing Project (PMP):** PMP is an ongoing and high visibility project. Many of the teams in MST-6 contribute either directly or indirectly to support this high-profile project. The project demands high quality and the deliverables are tracked frequently. *Customer: DOE NNSA defense program*
- Chemical Milling of Critical Shape: Chemical milling was used to produce a tapered stainless steel joint for a 100 Tesla research magnet at NHMFL. Multiple sheets were required with a tapered joint between sheets to prevent stress risers and failure. The size of the SS sheets made tapering by mechanical means impractical. Customer: National High Field Magnetic Laboratory
- Micro-Compression Pillars: Focused ion beam (FIB) was used to produce Cu-Nb and U-6Nb micro-compression pillars for mechanical testing. Customers: Global Nuclear Energy Partnership (GNEP), Enhanced Surveillance, DOE Office of Basic Energy Sciences, Los Alamos National Laboratory-Directed Research and Development (LDRD)
- Joint Test Assembly and Hydrotest: These projects involve high-profile deliverables and, at times, compressed schedules to enable quality testing. Many of the teams within MST-6 support these programs and allow deliverables to be met early. Rolling, punching, hydroforming, spin forming, and welding are a few of the processes used. *Customer: DOE NNSA defense program*
- High Performance Beryllium High Heat Flux Divertor: Prototypes were made using a unique beryllium plasma spraying system. A patent disclosure was filed for the technology which allowed deposition of 10 mm thick deposits. These prototypes passed severe high heat flux testing in the US and European Union and qualified the process for use in International Experimental Thermonuclear Reactor. Customers: DOE Department of Fusion Energy Sciences, European Fusion Development Association (EFDA)

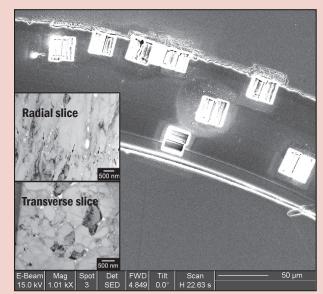


Gas metal arc welding in an inert chamber

Five-cell superconducting cavity: MST-6 personnel formed the 1/2 cells from high purity niobium sheet, vacuum brazed stainless steel flanges to niobium



tubes, electron beam welded the assembly, and chemically polished the inner surface to provide exceptional performance for a proton accelerator cavity. The capability to perform all these processes under one roof is unique. *Customer: Accelerator Production of Tritium project* 



National Ignition Facility (NIF) targets: The targets are 2 mm OD hollow spheres of beryllium alloyed with copper, to be filled with deuterium-tritium (DT) gas before use. The process developed by MST-6 to produce the alloy included arc-melting and casting plus equichannel angular extrusion (ECAE) with backpressure to refine the grain structure. Cast grain sizes of 2.5 mm were reduced to less than 30 μm using this technique. The focused ion beam was used to take samples out of the 150-μm thick walls of the NIF capsule. *Customer: NIF* 

#### Materials Science and Technology Division Facility Focus

#### **Materials characterization**

Characterization—a critical aspect of every materials program—is an important tool for understanding the properties and processing of materials and for applying that understanding to materials development. The Sigma Complex supports a collection of tools for characterizing materials including mechanical testing, hardness testing, electron microscopy, optical metallography, interstitial element analysis (O, N, C, H), trace element analysis using inductively coupled plasma, differential thermal analysis, thermogravimetric analysis, bulk density, surface area analyses, x-ray diffraction, and powder characterization capabilities.

#### **Recent projects**

- **Pit Manufacturing Project:** The large suite of optical microscopy techniques were used to certify that a piece of equipment involved in pit production is working within operational parameters. *Customer: NNSA defense program*
- Surface Damage in Beryllium: Orientation imaging microscopy (OIM) was used to quantify the amount of surface damage in Be from different machining techniques. *Customer: Weapons Engineering Technology Division*
- Mechanism for Uranium-Niobium Alloy Aging: Detailed examination of accelerated-aged specimen microstructures were made to clarify the exact aging mechanism and insure that the aging model had the correct scientific basis. In this study the distribution of Nb atoms and phases were probed with a variety techniques, each of which had its particular strengths. These included induction dilatometry, light and electron microscopy; and x-ray, neutron diffraction and three-dimensional atom probe tomography. Spatial resolution <1 nanometer needed to differentiate among several aging mechanisms that have been proposed. *Customer: Stockpile Stewardship* 
  - Hydrogen Interactions in Neutron Target Tube Loading: Valence

# The dual focused ion beam/high resolution scanning electron microscope, housed in Sigma's Electron Microscopy Laboratory, is used for a variety of projects, including producing micropillars for compression testing.

band x-ray photoelectron spectroscopy (XPS) was used to identify and characterize erbium oxide compounds found at preload, at post load, and at various stages of aging. Structure could be deduced from behavior observed at the Fermi edge. *Customer: NNSA Neutron Tube Target Loading* 

• Measuring Properties from Small Selected Areas: Successfully developed micro mini tensile specimen which allows measurement of properties in very small locations. *Customer: Pit Certification Program* 

#### **Modeling and process simulation**

Researchers at the Sigma Complex use fluid flow and heat flow computer codes to model microwave-material interactions, welding processes, rapid solidification by melt spinning, and the casting of near net shape components. They also use solid deformation modeling to examine sheet-forming processes and predict workpiece response to a number of different process variables. Scientists are involved with modeling and analysis of crystallographic texture development as a result of processing.

# Modeling of U6%Nb basic hemisphere castings showing fine detail of possible hot spots.

#### **Recent projects**

- **Robust Manufacturing:** The sensitivity of part properties to production parameter variations was modeled using the Truchas code. *Customer: Stockpile Stewardship*
- Optimum Design for Hydrotest Casting: The multi-physics code Truchas, developed under the ASC Telluride project, was used to simulate the performance of several mold designs for casting a hydro test part. This saved time and money. Customer: Testworks Program (part of an Enhanced Collaboration with the UK Atomic Weapons Establishment on radiation cases)
- Reliable Replacement Warhead (RRW): Modeling Components in 19 Days: The Los Alamos RRW design relied heavily on casting technology and modeling for a variety of components within pits, secondaries, and radiation cases. To meet the RRW hydro schedule, process modeling with the Truchas code was successfully utilized to develop casting parameters for the radiation midcase and resulted in an acceptable part 19 working days from receipt of the part definition. The short turnaround hinged on using simulation tools including Truchas and demonstrated a responsive capability for manufacturing never before seen in the Nuclear Weapons Complex. *Customer: NNSA*

#### **Materials Science and Technology Division Facility Focus**



#### **Synthesis and processing**

Over the history of the Sigma Complex almost every element on the periodic table between hydrogen and uranium has been handled or processed—and this capability still exists today. Since many of these elements have hazardous characteristics, this facility provides the appropriate safeguards for both personnel and environmental protection. The Sigma Complex provides an appropriate workplace for researchers who must work with a variety of materials, including beryllium, uranium, and thallium. Some of the processing techniques available at the Sigma Complex include induction heating or arc heating for alloy melting and casting; powder atomization and melt spinning; solution processes to synthesize ceramic powders or coatings; solution and plasma spraying to produce functionally gradient materials; slip casting, cold pressing and sintering, hot pressing, hot isostatic pressing; and microwave processing.

#### **Recent projects**

• Composite Material in Specialized Shapes:

Tungsten powder reinforced uranium 1/4 scale long rod penetrators with enhanced armor penetration and W-Ni-Fe 1/4 scale long rod penetrators were produced using hybrid processes. *Customer: DARPA Armor-Anti Armor Program* \* Transferred technology to Westinghouse, Union Carbide, and IIT

• New Large Format Radiation Detection

**Material:** Nanocrystalline LaBr<sub>3</sub> material was made in bulk quantities using rapid solidification and a novel consolidation technique for large format radiation detection scintillators. *Customer: Domestic Nuclear Detection Office (DNDO)* 

- Heavy Fermion Superconductors: Synthesis of single crystals of the actinide beryllides UBe<sub>13</sub> and ThBe<sub>13</sub> has been performed in beryllia crucibles by precipitation from molten metallic fluxes. The electronic properties of UBe<sub>13</sub> is thought to be related to the electronic properties of high temperature superconductors. Customer: DOE Office of Basic Energy Sciences
- Foams with Nanometer Features: A self-propagating combustion process for the synthesis of high surface area, low density metal foams has been discovered. To our knowledge, the observed surface area of the metal foams exceeds any reported in the literature. The process is in development for numerous metals including iron, cobalt, nickel, copper, palladium, platinum and

silver and for lithium nitride. The foams are being examined for uses in hydrogen storage and catalysis. *Customer: LDRD* 

#### SIGMA COMPLEX IN DETAIL

The Sigma Complex at Los Alamos National Laboratory totals more than 200,000 square feet of laboratory space, comprising three large facilities and several smaller ones. The complex, built in the 1950s and 1960s, houses extensive laboratory areas for materials synthesis and processing, characterization, and fabrication. Approximately 175 people work at the complex, including technical staff, technicians, administrative staff, and building support personnel. The Press Building (SM-35) has approximately 10,000 square feet. This building houses a 5,000-ton capacity hydraulic press with approximately 12 feet of maximum opening and also contains laboratory space available for hazardous materials research

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